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Effect of short-term ageing temperature on bitumen properties

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Properties of asphalt mixtures after ageing are fundamental parameters in determining long-term performance (e.g. durability) of these materials. With increasing popularity of reduced temperature mixtures, such as warm-mix asphalt, WMA, the question remains how a reduction in short-term ageing affects the properties after long-term ageing of bituminous materials. This paper aims to improve our understanding of the effect of asphalt manufacturing temperature on ageing and the resulting mechanical properties of bituminous binder by studying the effect of short- and long-term ageing of different bitumen samples as a function of short-term ageing temperatures. For this purpose, round robin experiments were conducted within the RILEM technical committee (TC) 252 chemo-mechanical characterisation of bituminous materials by 10 laboratories from 5 countries using four binders of the same grade (70/100 pen) from different crude sources. The short-term ageing was carried out using the standard procedure for rolling thin film oven test (RTFOT), but varying the temperatures. Long-term ageing was carried out using the standard procedure for pressure aging vessel (PAV) in addition to RTFOT. For the mechanical characterisation, rheological data were determined by using the dynamic shear rheometer (DSR) and conventional tests, with needle penetration and softening point using the ring and ball method. The results show that although different short-term ageing temperatures showed a significant difference in the mechanical properties of the binders, these differences vanished after long-term ageing with PAV.

Keywords: bitumen; DSR; long-term ageing; short-term ageing; asphalt; RTFOT; PAV

Introduction

As an organic material, bitumen undergoes changes in its mechanical characteristics, chemical composition and microstructure due to environmental effects over its lifetime (Glotova, Gorshkov, Kats, Shapiro, & Yureva, 1980; Lee, Tia, Ruth, & Page, 1997; Mills-Beale et al., 2012; Yao, Dai, & You, 2015). Loss of volatile components at elevated temperatures and oxidation are two main factors for these changes. In terms of mechanical properties, bitumen tends to become stiffer, more elastic and brittle over time. With regard to chemical composition, a shift within the

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SARA (saturates, aromatics, resins and asphaltenes) fractions can be observed with an increasing amount of asphaltenes and decreasing amount of aromatics over time (Hofko, Eberhardsteiner, Fussl, & Grothe, 2016; Lu & Isacsson, 2002). These changes are generally summarised under the term "ageing". Bitumen ageing can be split up into short-term and long-term ageing. In the case of hot mix asphalt, short-term ageing occurs during asphalt mix production, transport and compaction at the construction site, all of which happens within hours. It is characterised by relatively high temperatures (> 130°C) and thus, high oxidation rates. Long-term ageing occurs in asphalt pavements in the field as a result of exposure to traffic and climatic conditions during its service life. Compared to short-term ageing, long-term ageing is a slow oxidation process that occurs mostly in the top few millimetres of the surface course. Whether oxidant agents, other than atmospheric oxygen, play a role in long-term ageing, and how strong the impact of UV radiation on the ageing process is, are both still a subject for scientific debate (Hofko et al., 2015). While the effect of UV on asphalt ageing has been reported as early as half a century ago (Vallerga, Monismith, & Granthem, 1957), the existing standard ageing procedures ignore the effect of UV on asphalt ageing. One reason for this is that the UV effect applies to the upper surface of asphalt pavements only and thereby the effect on the total structure can be negligible (Durrieu, Farcas, & Mouillet, 2007). In terms of ageing simulation in laboratory, quite a few methods have been tried (Airey, 2003; Colbert & You, 2012). The most widely used are the rolling thin film oven test (RTFOT) to simulate short-term ageing and the pressure aging vessel (PAV) test to simulate long-term ageing. It was also reported that the ageing effect of UV and PAV in terms of the stiffness and phase angle depends on asphalt source and grade (Xiao, Amirkhanian, Karakouzian, & Khalili, 2015). Ageing of bitumen has a crucial effect on the durability of asphalt mixtures (Lu, Redelius, Soenen, & Thau, 2011), since stiffer and more brittle binders make an asphalt pavement more prone to cracking.

Warm-mix technologies are technologies to produce and compact asphalt mixtures at lower temperatures. They have been increasingly used in the asphalt industry around the globe over the last few decades. The main advantages of warm-mix asphalt (WMA) mixtures are lower energy consumption during mixing and compaction and therefore, lower greenhouse gas emissions (Kim, Lee, & Amirkhanian, 2010; Lee, Amirkhanian, Park, & Kim, 2009). They are seen as a more energy efficient, sustainable way for road construction (Hofko, Dimitrov, et al., 2016). In addition, WMA are also used in unfavourable weather situations (mainly low temperatures) to achieve a longer compaction phase at the construction site. An additional advantage of reduced temperature in production is reduction in harmful fume emissions. There are no conclusive results showing whether or not lower production temperatures, which lead to less oxidation during short-term ageing, also affect long-term ageing of binders and asphalt mixtures (Arega, Bhasin, & De Kesel, 2013; Banerjee, de Fortier Smit, & Prozzi, 2012; Menapace, Masad, Bhasin, & Little, 2015).

The question of how the chemical composition, the mechanical behaviour and microstructure of bitumen are linked has been of interest to researchers for decades (Eberhardsteiner et al., 2015a). An increasing number of studies related to this question have been observed in recent years (Allen, Little, Bhasin, & Glover, 2014; Eberhardsteiner et al., 2015b; Handle et al., 2016; Poulikakos et al., 2014). This may be related to the fact that advances in microscopy and spectroscopic techniques allow for a more detailed observation of complex organic materials like bitumen.

The RILEM technical committee (TC) 252 on chemo-mechanical characterisation of bituminous materials (CMB) is dedicated to answering some of the open questions in this field through round robin experiments. To this end, the experimental programme investigates the effect of production temperature on short- and long-term ageing of bitumen and tries to establish links between changes in the mechanical characteristics and chemical composition due to ageing. The round robin tests are to be finalised in 2016. The first results are presented in this paper.

Motivation and objectives

Since temperature has a significant effect on reaction kinetics in general and therefore on the oxidation rate, lower production temperatures of asphalt mixtures lead to less short-term ageing of bitumen. However, it is not clear whether this effect can also be observed in reduced long-term ageing. In addition, extensive data and knowledge have been gathered on how ageing influences bitumen in terms of its mechanical behaviour and its chemical composition. However, these data have mostly been built up separately for mechanical testing and chemical analysis.

The main objective of this paper is to present the first results of the study on short- and long-term ageing of different bitumen samples from a variety of crude sources, varying short-term ageing temperatures. The virgin, short-term and long-term aged samples are evaluated by means of different mechanical and chemical analysis methods. The focus of this paper is the results of the mechanical analyses. Effort is made to increase the understanding of short- and long-term ageing and the impacts of short-term ageing temperature on the evolution of bitumen properties.

Materials and methods

A group of 10 laboratories from 5 countries, from both academia and industry, worked together in the round robin experiment. The laboratories involved were the Technical University of Vienna in Austria, Empa in Switzerland, University of Braunschweig in Germany, Arizona Chemical in the Netherlands, IMP in Switzerland, Nynas in Sweden, Michigan Technical University in the USA, Q8 in the Netherlands, Repsol in Spain and the University of Nottingham in the UK.

Binder samples

Four bituminous binders, which were 70/100 pen according to EN 12591 from different crude sources were used in the overall study. They were labelled B501, B502, B503 and B504. Through preliminary tests, it was seen that there were two families of binders in this group. This paper focuses on the results of binders B501 and B504. In addition, suffixes A, B_Temp and C_Temp are used to give information on the state of ageing and the ageing conditions as follows:

- Unaged binder: A (e.g. B501A)
- RTFOT-aged binder: B_Temp (e.g. B501B_163 for sample B501 aged in the RTFOT at 163°C)
- PAV-aged binder: C_Temp (e.g. B501C_143 for sample B501 aged in the RTFOT at 143°C and subsequently in the PAV)

Experimental methods

A detailed programme and work instructions have been developed and used by all laboratories as shown in Figure 1. It consists of three parts, differing in the conditioning temperature for the RTFOT, 163°C for Part A, 143°C for Part B and 123°C for Part C. For all virgin, short-term and long-term aged samples, DSR testing, as well as penetration and softening point (ring and ball) were carried out. These results are the topic of this paper. In order to characterise the ageing chemically, as indicated in Figure 1, Fourier transformed infrared spectroscopy (FTIR) were also run on all samples. These ongoing analyses will be reported on in the near future.

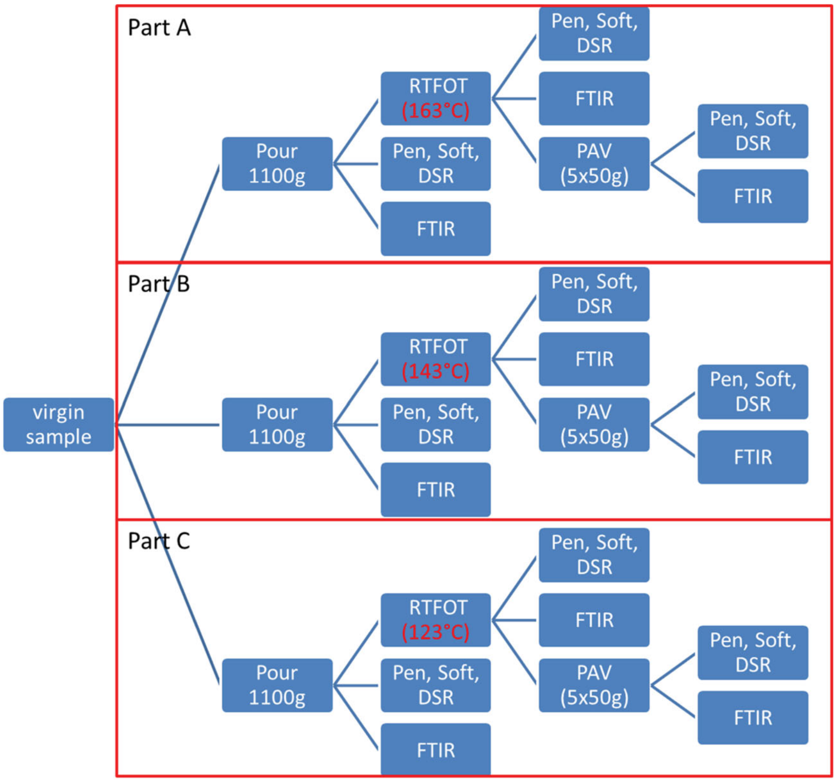

Figure 1. Outline of the test programme.

Short-term ageing was simulated using the RTFOT. In this study, the test was carried out in accordance with EN 12607-1 for 75 min and at three different temperatures 163°C, 143°C and 123°C, which covers temperatures used for hot and warm applications.

Long-term ageing was simulated using the pressure ageing vessel (PAV) test. This mimics ageing on the road during the life time of the pavement. In this study, the test was carried out in accordance with EN 14769 after RTFOT. Tests were conducted at a temperature of 100°C and an air pressure of 2.1 MPa for 20 h.

To relate to the different ageing effects with standard tests, conventional bitumen testing was performed. These tests were the softening point test according to EN 1427 which represents the consistency at high temperatures and the penetration test at 25°C according to EN 1426, which represents the consistency at ambient temperature.

To fully characterise the mechanical properties, the dynamic shear rheometer (DSR) was used to measure the rheological behaviour of bituminous binders under oscillatory sinusoidal loading. Therefore, linear viscoelastic parameters (Aklonis & MacKnight, 2005; Christensen, 2010) such as complex shear modulus $|G^*|$ and phase angle δ, can be obtained over a wide range of temperatures and frequencies, based on a standard testing procedure. In the present study, most of DSR testing was made with a parallel-plate geometry with an 8 diameter and 2 mm gap for temperatures between -10°C and $+40$°C and a 25 diameter and 1 mm gap from $+30$°C to $+80$°C. Tests were carried out with temperature steps of 10°C and a frequency sweep from 0.1 to 10 Hz at each temperature.

Results and discussion

Conventional bitumen characteristics

Traditional penetration value at 25°C and softening point temperature were used to monitor the ageing evolution of the binders from their initial condition through RTFOT ageing at 163°C, 143°C and 123°C and subsequent PAV ageing. Penetration is probably the simplest measure of bitumen's properties. As bitumen is aged, its penetration decreases.

Figure 2 shows the results of the penetration change for the B501 and B504 binders. This graph shows the average results from all laboratories along with the standard deviations for each condition. Initially the unaged bitumen has a penetration of just below 80 × 0.1 mm. The effect of RTFOT ageing at different temperatures can be clearly seen. Increasing RTFOT temperature increases the degree of ageing. RTFOT at 163°C reduces the average value of penetration to just below 50 × 0.1 mm, RTFOT at 143°C to approximately 60 × 0.1 mm and RTFOT at 123°C to about 70 × 0.1 mm. However, after subsequent PAV ageing, all RTFOT conditions had their average penetration value reduced to approximately 30 × 0.1 mm. This shows that the effect of PAV aging is more severe than RTFOT ageing. After PAV ageing the penetration of the sample RTFOT aged at 123°C was approximately 31–32 × 0.1 mm, whereas the PAV-aged samples which were RTFOT aged at 143°C and 163°C had penetrations of approximately 28–29 × 0.1 mm. As shown in Figure 2, while there is still a difference in penetration results between RTFOT temperatures, these differences are limited after PAV ageing.

Figure 3 shows the results for the softening point temperature as a function of RTFOT ageing temperature for the B501 and B504 binders after different ageing steps. Initially the unaged bitumen had a softening point of 46°C. After RTFOT ageing at 123°C the softening point increased to 48°C, after RTFOT at 143°C the softening point rose to about 50°C and after RTFOT at 163°C to just above 51°C. After PAV ageing the differences in softening point between the samples were reduced. However, PAV ageing after RTFOT at 123°C raised the softening point to about 57°C in the case of both the B501 and B504 binders. PAV ageing after both RTFOT at 143°C and 163°C raised the softening point to about 58°C and 59/60°C for the B501 and B504 binders, respectively. So although differences in ageing were diminished, RTFOT at 123°C could still be distinguished after PAV ageing. The standard deviation of the results after PAV ageing was higher than after RTFOT ageing. Thus, the ageing temperature used for RTFOT has no significant impact on softening points after PAV ageing. The softening point evolution, again, generally shows that, differences in ageing after RTFOT at different temperatures are generally offset by the subsequent PAV ageing.

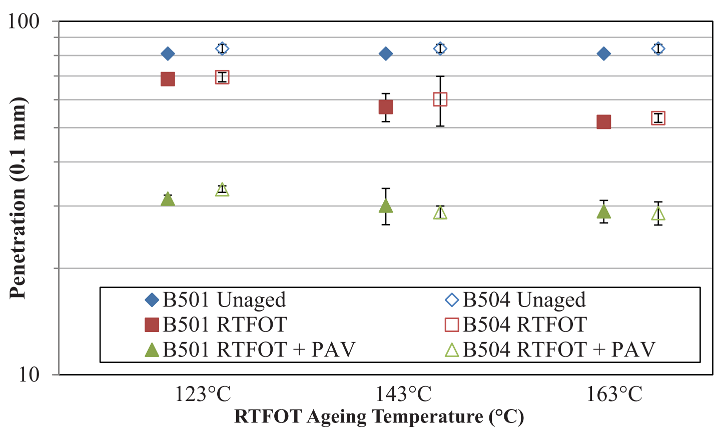

Figure 2. Penetration value as a function of RTFOT ageing temperature for bitumen B501 and B504.

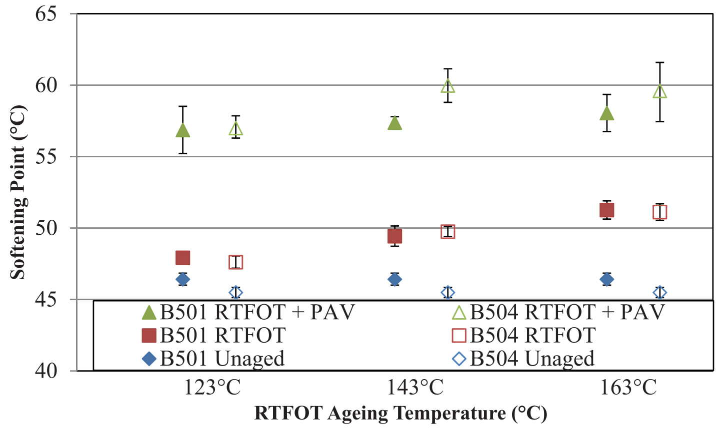

Figure 3. Softening point as a function of RTFOT temperature for bitumen B501 and B504.

Dynamic shear rheometer

The data obtained from the DSR in frequency sweep tests were analysed using complex modulus, |*G**|, and phase angle, δ, with master curves produced based on the time-temperature superposition principle. The curves were generated according to the procedure proposed by (Bahia, Hanson, Zeng, & Zhai, 2001) for all the seven different ageing conditions investigated in this research. Figures 4 and 5 provide |*G**| master curves for asphalt binders B501 and B504 obtained by one of the participating laboratories as an example.

Both plots present similar trends. A difference between short- and long-term-aged materials can be clearly observed. At higher frequencies corresponding to lower temperatures, the absolute value of the complex modulus converges towards a common threshold regardless of the ageing conditions. At lower frequencies and higher temperatures, some differences can be visually detected between the different ageing conditions for the two ageing groups, RTFOT and RTFOT + PAV, respectively. Nevertheless, it is not uncommon to observe larger differences in the values of |*G**| when restricting the analysis to the highest testing temperature in

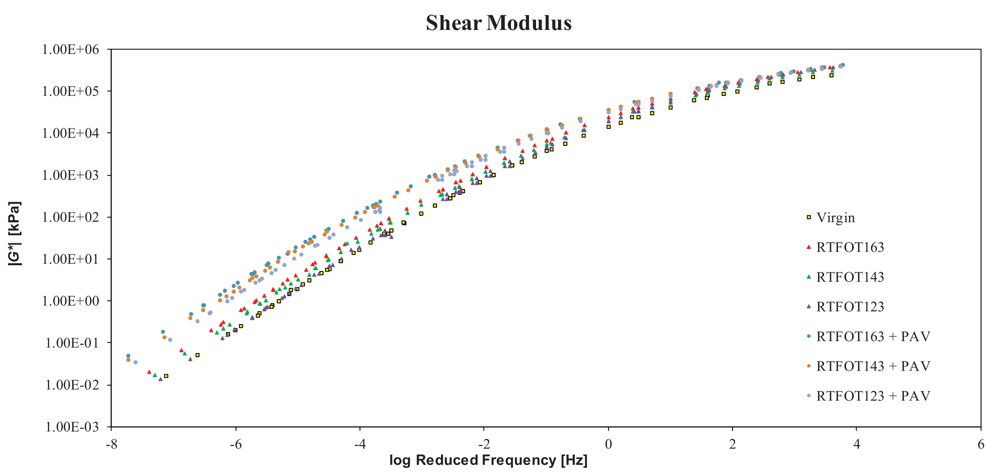

Figure 4. Complex shear modulus master curves for asphalt binder B501 at reference temperature of 10°C.

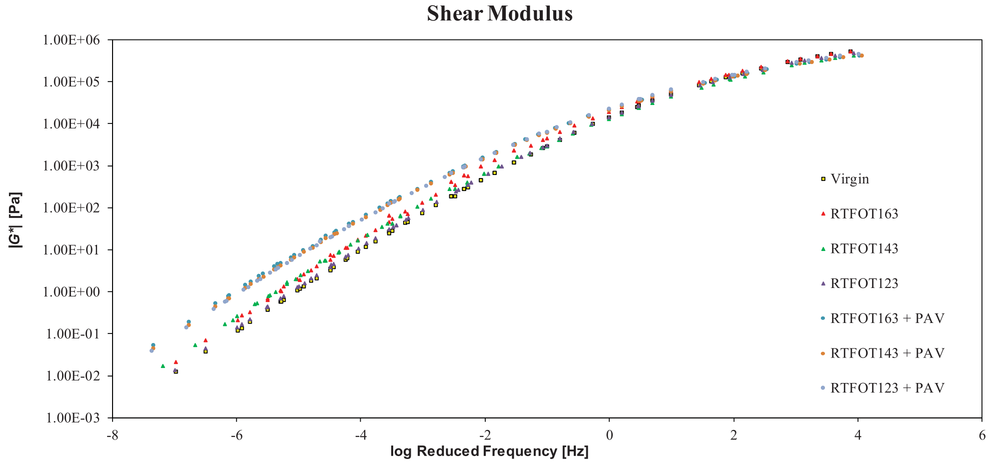

Figure 5. Complex shear modulus master curves for asphalt binder B504 at reference temperature of 10°C.

correspondence to lower frequencies. When comparing the two master curves it seems apparent that the two binders show a different ageing pattern across the frequency spectrum.

In order to quantitatively verify the significance of the visual analysis performed on the master curve, bar charts were prepared using the data generated by four laboratories. For this purpose, the analysis of $|G^*|$ and δ was restricted to four specific temperatures, -10°C, 0°C, 40°C and 70°C and to one single frequency, 1.59 Hz (10 rad/s), as used in the superpave performance grade

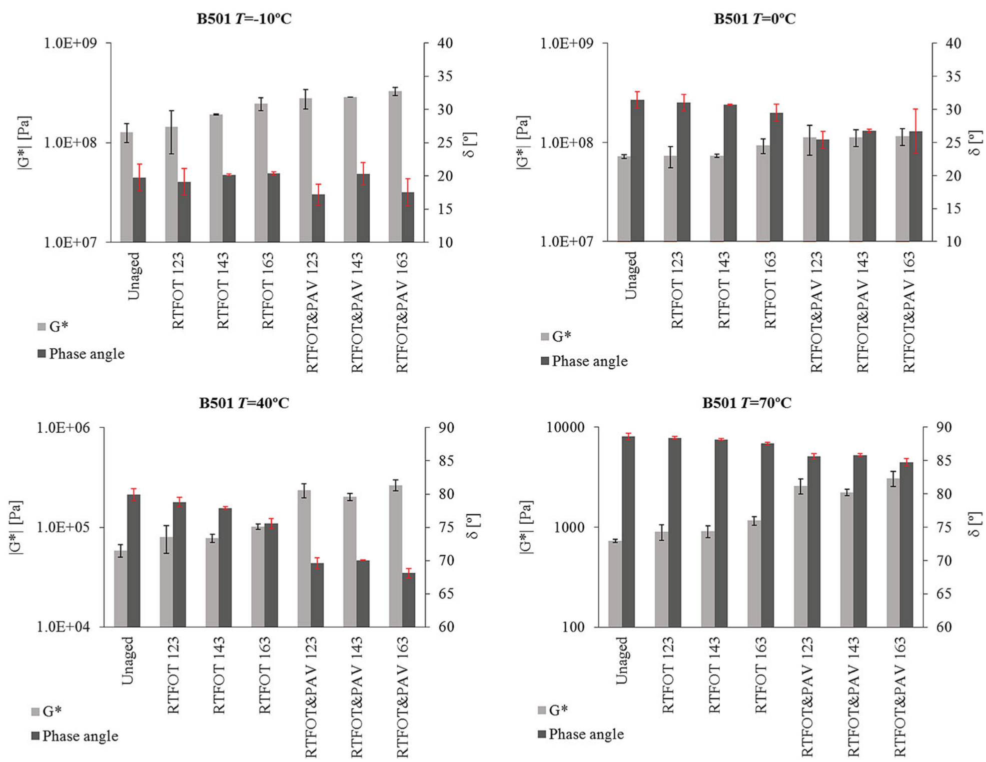

Figure 6. Complex modulus and phase angle of asphalt binder B501.

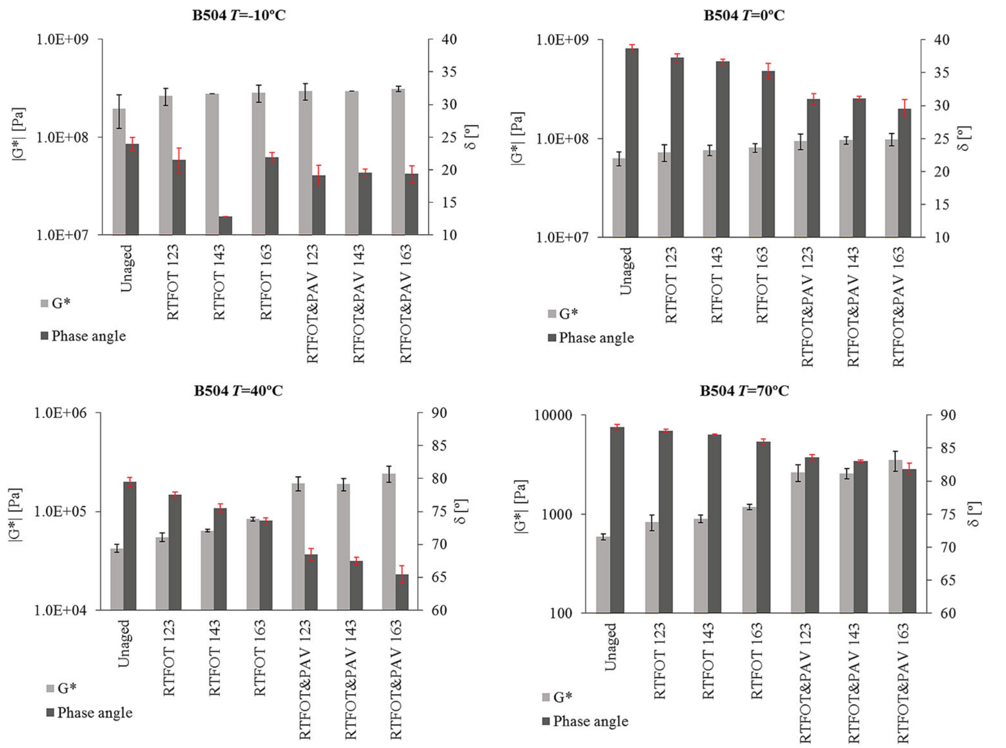

Figure 7. Complex modulus and phase angle of asphalt binder B504.

approach. From an initial analysis larger differences in both in $|G^*|$ and δ were observed among laboratories. This may be due to, amongst other things, testing device, operator and testing mode (strain and stress controlled). An additional explanation can also be linked to the four specific temperatures selected for the analysis. Although they cover the overall spectrum of temperatures selected for DSR testing, they cannot fully describe the evolution of $|G^*|$ and δ as a master curve would, hence being more prone to larger differences between the single measurements. Figures 6 and 7 present the bar charts of $|G^*|$ and δ for binders B501 and B504 at all the four selected temperatures.

A visual analysis of the bar charts suggests that a global stiffening of the material occurs across ageing conditions, from the unaged to the apparently more effective combination of RTFOT ageing at 163°C and PAV. This effect is more significant at higher testing temperatures (40°C in Figures 6 and 7). Within the different RTFOT temperatures, it appears that when using 163°C a more severe increase in complex modulus occurs compared to the other two temperatures: 143°C and 123°C. A similar trend can also be observed for the three corresponding ageing conditions when PAV is performed. Temperature seems to significantly affect the evolution of $|G^*|$ and δ and, hence, the actual ageing effectiveness. The testing condition at low temperature minimises the effect of ageing in reducing the differences in complex modulus.

As may be expected the phase angle increases at higher testing temperatures. Ageing seems to induce a significant decay in the value of δ only when PAV is performed, indicating the loss of the viscous component of the complex modulus, although a clear pattern cannot be identified. A more detailed statistical analysis will be performed in the progress of the research to quantitatively verify the visual analysis.

Summary and conclusions

The work presented in this paper shows preliminary results of a broad study carried out under the RILEM TC 252 CMB. It focuses specifically on the influence of short-term ageing temperatures, as used for WMA, on long-term ageing properties of bitumen. For this purpose, four different bituminous binders of same grade but different crude sources were tested using neat bitumen, RTFOT conditioning at different temperatures and followed by PAV conditioning. The results of the mechanical properties are presented; while chemical property evaluation including infrared spectroscopy is ongoing and will be presented at a later date.

The analysis of basic properties with penetration value at 25°C and softening point temperature has shown a difference after RTFOT with different short-term ageing temperatures. This difference vanishes completely for the penetration value and to a lesser degree for the softening point after PAV ageing.

The analysis of rheological properties over a wide range of temperatures and frequencies was conducted through DSR testing by looking at the complex shear modulus and phase angle. The results confirm initial trends from the basic properties of penetration and softening point. These show a more significant increase in complex modulus at higher testing temperatures, corresponding to low frequencies. Also ageing seems to induce a significant reduction in the phase angle after long-term ageing with the PAV.

The overall outcome showed that short-term ageing through RTFOT has a lower impact on binder properties with lower temperatures. However, after PAV ageing, which induces more severe ageing, that almost offsets the differences from short-term ageing. With further analysis of the chemical modifications resulting from aging and the effect of warm mix technologies on aging, it will be possible to get a better understanding of the overall benefits of these technologies. Furthermore, it is to be noted that the RTFOT temperature at 163°C does not corroborate the standardised mixing temperature that is dependent on bitumen grade, a point that warrants further thoughts on the appropriate RTFOT temperature.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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